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Nuclear reaction analysis as a technique of measurement of some oxide layer thicknesses

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Abstract : Nuclear reaction analysis (NRA) has been carried out through (d,p) reaction for evaluating oxide layer thicknesses with 1 MeV deuterons. Oxides of silicon and tantalum of layer thicknesses up to 13000 Å were investigated using integrated charge and beam current respectively 30 μC and 100 nA. The technique is found to be excellent for relative measurements of layer thicknesses. A calibration curve for future measurements of thickness of thin films of silicon and tantalum oxides is provided. The results so obtained are then compared with those of Rutherford backscattering spectrometry (RBS) technique.

Keywords : Measurements of oxide layer thickness, application of nuclear reaction analysis, surface barrier detector, (d,p) reaction

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1. Introduction

Over more than a decade, charged particle nuclear reactions and scattering methods have become increasingly popular in depth profiling of different nuclides in various materials. It is an established fact that the composition and thickness of oxides and nitride films on bulk elemental surface can be evaluated using NRA technique [1–6]. The technique has been developed for near surface analysis during the past few decades [1,7–14]. Experimentally, a light ion beam taken from a particle accelerator is incident on the surface of a target resulting in nuclear reactions of different types depending on the nature and energy of the ions and the nature of the target. Identification and depth profiling of the elements making up the target can be made. Using NRA technique concentration profiles over a few microns of the surface with resolution of the order of 1000 Å can be attained where depth resolution attainable is better than 200 Å [3,6].

Side by side, the application of RBS for near surface analysis has been developed during the past three decades. It is mainly applied in the semiconductor field and has been extensively applied in investigating silicon and its compounds. The method has taken a good place besides other modern surface analytical techniques such as secondary ion mass spectrometry (SIMS), auger electron spectrometry (AES) and X-ray fluorescence (XRF).

In this work, (d,p) reaction has been used in evaluating the layer thickness of some oxide samples. The reaction is favoured because of its general applicability and also since it provides a useful method of analysis of reactions involving all the major isotopes of elements having $Z = 4$ to $Z = 17$ which have positive Q values of the order of few MeV [15] thus making multielemental analysis possible. The results obtained from the (d,p) reaction analysis are then compared with those obtained through RBS technique on the same samples.

2. Principles

If we consider a thin and single element target on which a beam of ions with energy suitable to overcome the Coulomb barrier is allowed to incident (Figure 1a) then some of these ions

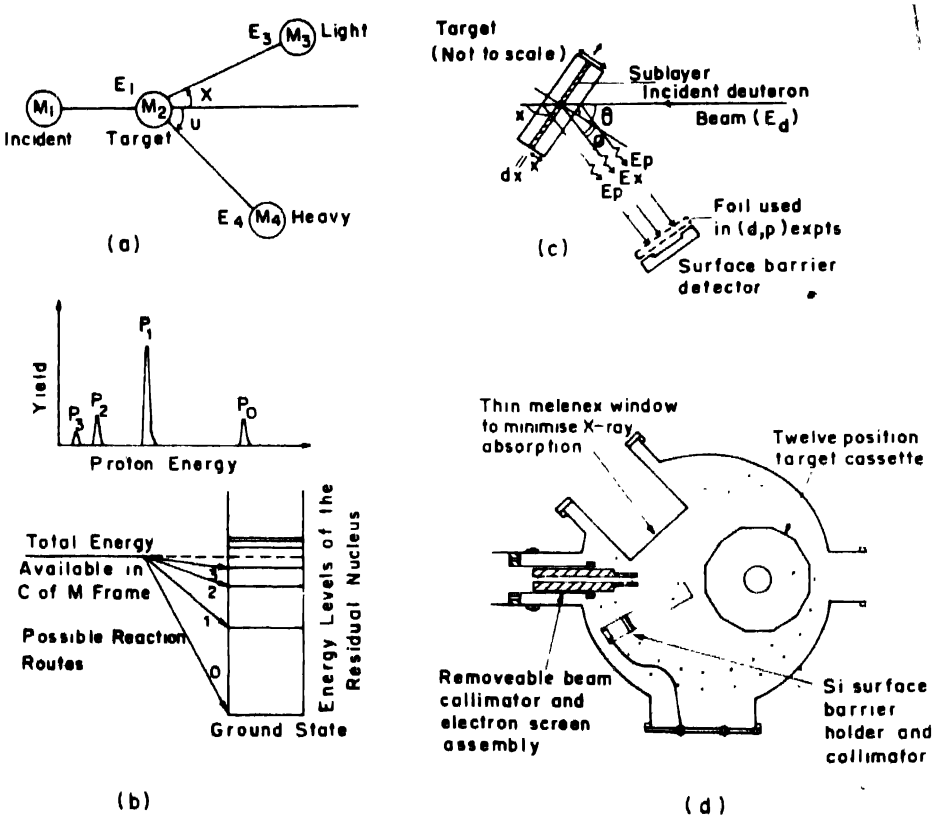

Figure 1. (a) Mechanism of a nuclear reaction. (b) The yield/energy spectrum of emergent protons at the detector. (c) Position of the detector with respect to the incident deuteron beam. (d) Schematic diagram of general purpose nuclear analysis target chamber.

undergo nuclear reactions with the target atoms. The energy of the light particles emitted from the reaction can easily be evaluated from the standard kinematic considerations [16].

The yield/energy spectrum of emergent protons that results when a large number of deuterons are incident on a thin target is an shown in Figure 1b. Any particular incident ion-target nucleus interaction can give rise to a series of peaks due to the different possible excited states of the daughter nucleus (Figure 1b). A calibration curve between emitted particle energy and peak channel number is sketched to identify unknown peaks. The usual way to reveal the depth profiles from the spectrum is to create a depth scale from stopping power tables or from the parameterized stopping powers including the effect of any detector foil.

3 Experimental

The $^{16}O(d,p)^{17}O$ reaction has been used to evaluate the oxide layer thicknesses of the samples cited in the Table 1. The experiment was carried out using the 3 MV Dynamitron accelerator

Table 1. List of the samples

Sl. no.	Name of the source	Composition	Quoted layer thickness (A)	Integrated area under $^{16}O(d,p_0)^{17}O$ peak	Calculated layer thickness from RBS
1	Plessey Ltd	SiO_2	1000	499 ± 28	1040 ± 60
2	Plessey Ltd	SiO_2	13504	5750 ± 81	13500 ± 180
3	GEC	SiO_2	647	323 ± 22	640 ± 60
4	GEC	SiO_2	1030	514 ± 29	1040 ± 60
5	GEC	SiO_2	2590	1294 ± 37	2600 ± 120
6	GEC	SiO_2	4007	2050 ± 49	4020 ± 180
7	Chalk River	Ta_2O_5	707	353 ± 22	710 ± 40
8	Chalk River	Ta_2O_5	1741	870 ± 31	1760 ± 40
9	Chalk River	Ta_2O_5	5435	2716 ± 51	5470 ± 80

All the samples are made of oxide layer on bulk silicon substrate.

at Birmingham Radiation Center, University of Birmingham. The geometries investigated to compare the depth resolution attainable using the $^{16}O(d,p)^{17}O$ reaction is similar to that described previously by Simpson *et al* [17]. Only the angle 135° has been considered in this work (Figure 1c).

Among the samples used in the work two oxide samples known to be SiO_2 were supplied by Plessey Ltd and four oxide samples all known to be the same composition were provided by GEC Research Laboratories (Hirst Research Centre). Three other oxide samples known to be Ta_2O_5 were obtained from Chalk River, Canada. All the samples possess their quoted oxide layer thicknesses as mentioned in the fourth column of Table 1. The Ta_2O_5 films were developed on tantalum and SiO_2 on silicon. 1 MeV deuteron beam was allowed to impinge on the samples mounted on the target holder (cassette) at 30° apart from each other carefully in such a way that the incoming beam strikes these sides of the samples normally (Figure 1d). A surface barrier detector (S 199) with depletion layer 700 μm was placed at the

said angle *e.g.* 135° to the incident beam direction. Beam current used was 100 nA and integrated charge was 30 μC. A foil of thickness 25 μm was used to stop the alpha particles which also come out of this reaction. The beam collimation and detector bias used were respectively 1 mm and 200 volts. The main amplifier gains were as follows :

Course gain = 200
Fine gain = 0.9
Shaping time = 0.25 μsec

The beam tube and the target chamber were evacuated by vacuum pumps to 10^{-6} Torr. The targets (samples) were striken by the incident beam of deuterons one after another normal to the plane of the target surface. Signals from the detector were fed by short cables to an Ortec 125 pre-amplifier and than to an Ortec 472A main amplifier by a long cable at counting station. The resulting pulses were passed in turn to a Hewlet Packard (HP) 5416B ADC and 2100 HP Computer Multichannel Analyzer for the spectrum storage and display. The spectrum obtained was stored on the disk and then copied to the magnetic tape for analysis.

For (d,p) reaction, generally, an absorber foil thick enough to stop elastically scattered deuterons from the target but thin enough to allow the expected reaction product (protons) to pass through is placed over the detector. This reduces the pile up effect and hence increases the sensitivity of the technique.

4. Results and discussion

Figures 2 and 3 show two of the yield/energy spectra obtained in this experiment. The highest energy group *e.g.* the $^{16}O(d,p_0)^{17}O$ arising from the $^{16}O(d,p)^{17}O$ reaction has been considered

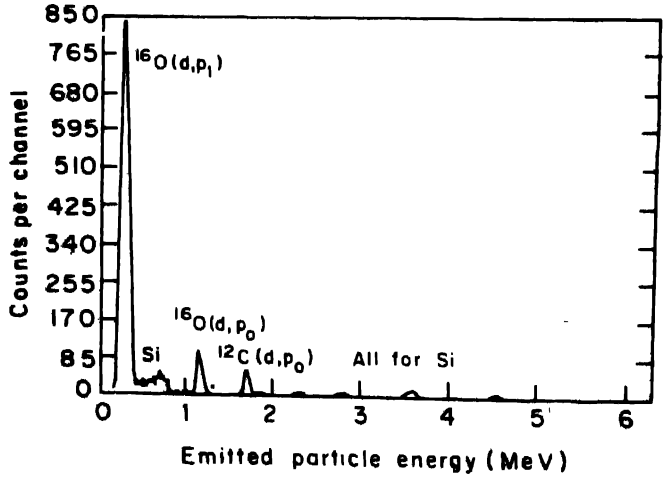

Figure 2. Energy spectrum of the (d,p) reaction for 1 MeV deuteron on SiO_2 (GEC) sample having quoted layer thickness 2590 Å.

only. Other groups were contaminated by Si and Ta groups and hence were avoided. Integrated area of the (d,p_0) peak of each spectrum has been calculated. Figure 4a is a plot

showing the variation of the integrated areas with respect to the quoted oxide layer thickness of the samples. In the work the cross section was supposed not to very appreciably over the

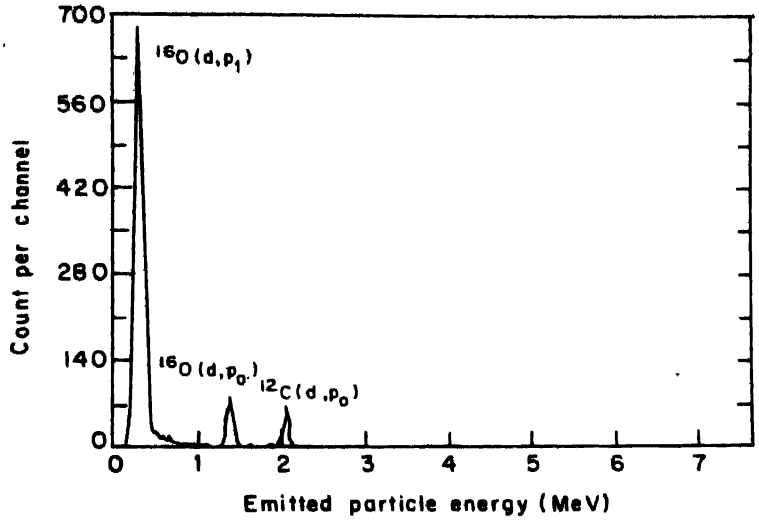

Figure 3. Energy spectrum of the (d,p)reaction for 1 MeV deuteron on Ta_2O_5 (Chalk River) sample having quoted layer thickness 1741 Å

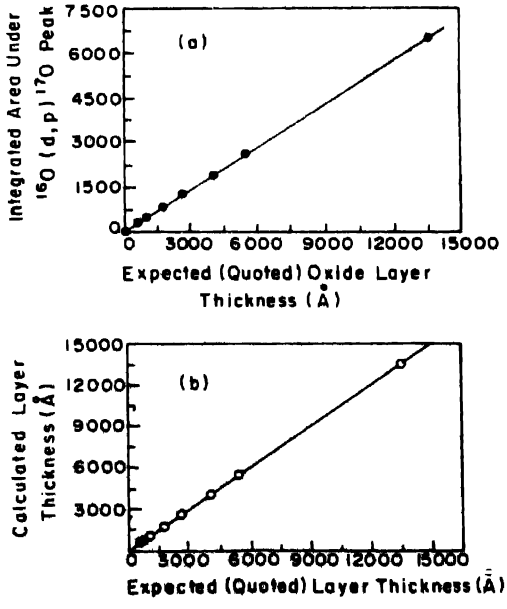

Figure 4. (a) Plot of expected layer thickness *versus* integrated area under $^{16}O(d,p_0)^{17}O$ peak for 1 MeV deuteron. (b) Plot of calculated layer thickness *versus* expected layer thickness for RBS analysis.

range of energy loss of the incident particles in the layer being investigated. Hence the total reaction yield gives a measure of the number of the atoms present and the energy distribution gives a measure of the depth profile.

The variation shows a straight line passing through the origin. The peak areas depend on charge integration, cross section for this reaction, geometry, experimental conditions etc. The cross section is not well known for this reaction. Hence we can not get absolute thicknesses using this curve, rather only the relative values of thickness may be obtained.

Column 6 of the Table 1 shows the calculated layer thickness extracted from RBS experiment on the same oxide samples measured in a previous study [18] by us. The corresponding graph showing the variation of calculated layer thicknesses with respect to the quoted layer thicknesses is shown in Figure 4b. The graph shows that an absolute measurement of oxide layer thickness is possible using RBS technique because the curve passing through the origin maintains a slope 1 while showing variation of calculated layer thickness with respect to the quoted layer thicknesses. However for a much thicker layer RBS technique would face difficulty in extraction of depth profiles, where (d,p) reaction analysis method may still be useful.

It seems worthwhile to make a comment here about suitability of NRA as compared to other techniques *e.g.* SIMS, AUGER etc. NRA is a non destructive technique. It has the additional advantage that the analysis time is usually quite short, of the order of 10 minutes per sample. NRA technique can be used as a very useful alternative to SIMS and AUGER which involve sputtering to provide depth information. NRA is thus a better method than SIMS and AUGER at least in one aspect *i.e.*, it is free from artifacts inherent in methods involving sputtering.

5. Conclusions

The $^{16}O(d,p)^{17}O$ reaction has been used for relative depth measurement on oxide layers. NRA technique using $^{16}O(d,p)^{17}O$ reaction cannot be employed for absolute depth measurements, where as RBS techniques can be employed for such a purpose. On the other hand NRA technique can be employed for a thicker layer where RBS technique would fail. A few additional advantage of NRA is that it is an non-destructive technique and allows quite a short time analysis.

We have not got reliable data above 13000 Å for oxide samples. It is felt that such data and also oxides of materials other than silicon and tantalum would be useful for added check.

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